Chemical Characterization of SiGe Single-Crystal Specimens and SiGe Films on Silicon with Electron Probe Microanalysis

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The CSTL-led research team provides the semiconductor industry with a reference material for evaluation of SiGe components.

The research team investigated three aspects of SiGe thin-films in an effort to prepare reference materials of SiGe on Si. First we studied thin films of SiGe on 8-inch diameter Si wafers for heterogeneity using wavelength dispersive electron probe microanalysis (WD-EPMA). Twelve 2 cm x 2 cm specimens obtained from 2 different wafers were studied. The nominal compositions of the two films were 0.0384 and 0.341 mass fraction Ge, respectively; their approximate thickness was 95 nm and 170 nm, respectively. Secondary-ion mass spectrometry (SIMS) and elipsometry revealed a circular halo-like region around the disk center that was thicker than for the rest of the disk thus suggesting specimen-to-specimen heterogeneity. With this knowledge, specimens were selected from a region in the wafer where the specimen-to-specimen thickness variation was expected to be minimal. Using a point beam – taking readings on 10 points per specimen – the extent of the point-to-point heterogeneity within each specimen and between specimens was assessed. The expanded uncertainty, including all heterogeneity contributions, was estimated to be no larger than 2.5% for Ge in 10 specimens.

A heterogeneity assessment was conducted using WD-EPMA of five specimens cut from each of the SiGe14 and SiGe6.5 standard boules which were previously assessed for heterogeneity in 2003. The Ge in these five specimens had been analyzed with instrumental neutron activation analysis (INAA) and a sixth specimen had been analyzed by inductively-coupled plasma optical emission spectrometry (ICP-OES). We determined that the same sample-to-sample trends in the Ge composition shown by INAA were observed by WD-EPMA. Data, including backgrounds, were acquired at three different excitation potentials to enable quantification and evaluation of matrix correction procedures. This evaluation is ongoing and seeks to compare its results with those obtained using INAA and ICP-OES.

SiGe films on Si (approximately 4 μm thick) were prepared by ASM America. Given their thicker profile it is be possible to treat them as bulk specimens with regard to quantitative electron microprobe analysis. Four specimens cut from two different disks with nominal compositions of SiGe10 and SiGe25 were tested using traverses and duplicate readings on random points to assess the heterogeneity. In addition, we acquired data at three excitation potentials with background readings for quantification. Using all these data we found that the expanded uncertainty (k=3 or 99% confidence interval) is less than 1% relative for both Si and Ge, which is only slightly greater than the counting statistics error predicted from Poisson statistics. Currently, evaluation of the random point heterogeneity data and the quantification procedures are in process. Data from the standard boules will be used in conjunction with Monte Carlo calculations to determine the optimum voltage and corrections procedures for microprobe analysis of the thicker films. We will also continue to evaluate procedures for the quantitative electron probe analysis of the thinner films discussed in the first part of these studies.

CSTL plans to release the materials for distribution as "Interactive Materials"* during the first half of FY05.

***Interactive Materials** are materials that are donated by interested parties and distributed by NIST to private sector analysts. Data is submitted to the NIST IM Website, and evaluated by NIST staff that post collective results. This approach was designed to quickly make new materials (with some pedigree and evaluation) available to a specific community.